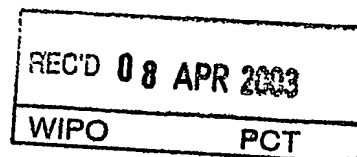




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the documents attached hereto are true copies of the Forms P2, P6,  
provisional specification and drawings of South African Patent

Application No. 2002/1428 in the name of ELEMENT SIX (PTY) LTD

Filed : 20 February 2002  
Entitled : Metal Coated Abrasive  
particles

Geteken te  
Signed at

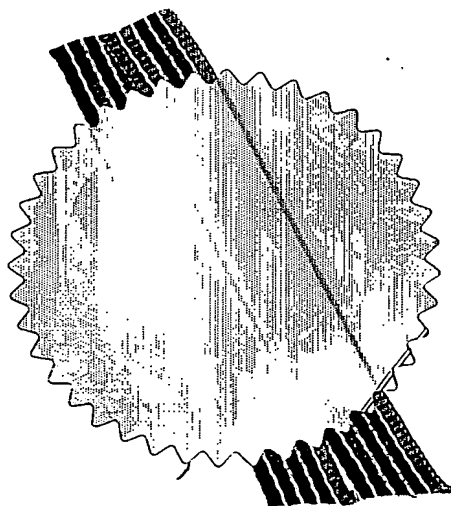
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in die Republiek van Suid-Afrika, hierdie  
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dag van  
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OFFICIAL APPLICATION		LODGING DATE: PROVISIONAL		ACCEPTANCE DATE		
21	01	22	20 FEBRUARY 2002	47		
INTERNATIONAL CLASSIFICATION		LODGING DATE: COMPLETE		GRANTED DATE		
51		23				
FULL NAME(S) OF APPLICANT(S)/PATENTEE(S)						
71	DE BEERS INDUSTRIAL DIAMONDS (PROPRIETARY) LIMITED ELEMENT SIX (PTY) LTD <div style="position: absolute; top: 0; right: 0; border: 1px solid black; padding: 2px;">           NAME VERANDER            NAME CHANGED         </div>					
APPLICANTS SUBSTITUTED:				DATE REGISTERED		
71						
ASSIGNEE(S)				DATE REGISTERED		
71						
FULL NAME(S) OF INVENTOR(S)						
72	PIPKIN, NOEL JOHN					
PRIORITY CLAIMED		COUNTRY	NUMBER	DATE		
N.B. Use International abbreviation for country (see Schedule 4)		33	NIL	31	NIL	
				32	NIL	
TITLE OF INVENTION						
54	METAL COATED ABRASIVE PARTICLES					
ADDRESS OF APPLICANT(S)/PATENTEE(S)						
SEO BUILDING, CORNER CROWNWOOD & BOOYSENS RESERVE ROADS, THETA, JOHANNESBURG, 2001, GAUTENG, SOUTH AFRICA						
ADDRESS FOR SERVICE				S & F REF		
74	SPOOR & FISHER, SANDTON			PA132492/P		
PATENT OF ADDITION NO.			DATE OF ANY CHANGE			
61						
FRESH APPLICATION BASED ON			DATE OF ANY CHANGE			

## APPLICATION FOR A PATENT

AND ACKNOWLEDGEMENT OF RECEIPT  
(Section 30 (1) - Regulation 22)

HASR 505

R 006000

The granting of a patent is hereby requested by the undermentioned applicant on the basis of the present application filed in duplicate

OFFICIAL APPLICATION NO.

21	01	2002/1428
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REPUBLIC VAN SUID AFRIKA  
S & F REFERENCE

PA132492/P

FULL NAME(S) OF APPLICANT(S)

71	DE BEERS INDUSTRIAL DIAMONDS (PROPRIETARY) LIMITED ELEMENT SIX (PTY) LTD 27.01.03
----	--

ADDRESS(ES) OF APPLICANT(S)

	SEO BUILDING, CORNER CROWNWOOD & BOOSENS RESERVE ROADS, THETA, JOHANNESBURG, 2001, GAUTENG, SOUTH AFRICA
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TITLE OF INVENTION

54	METAL COATED ABRASIVE PARTICLES
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THE APPLICANT CLAIMS PRIORITY AS SET OUT ON THE ACCOMPANYING FORM P.2. THE EARLIEST PRIORITY CLAIM IS:

COUNTRY:	NIL	NUMBER:	NIL	DATE:	NIL
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THIS APPLICATION IS FOR A PATENT OF ADDITION TO PATENT APPLICATION NO.

21	01	
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THIS APPLICATION IS A FRESH APPLICATION IN TERMS OF SECTION 37 AND IS BASED ON APPLICATION NO.

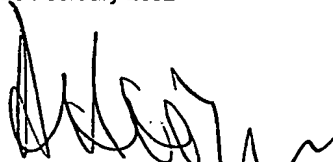
21	01	
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THIS APPLICATION IS ACCOMPANIED BY:

- ☒ 1. A single copy of a provisional specification of 8 pages.
- ☐ 2. Drawings of sheets
- ☐ 3. Publication particulars and abstract (Form P.8 in duplicate).
- ☐ 4. A copy of Figure of the drawings (if any) for the abstract.
- ☐ 5. Assignment of invention.
- ☐ 6. Certified priority document.
- ☐ 7. Translation of the priority document.
- ☐ 8. Assignment of priority rights.
- ☐ 9. A copy of the Form P.2 and the specification of S.A. Patent Application No .
- ☐ 10. Declaration and power of attorney on Form P.3.
- ☐ 11. Request for ante-dating on Form P.4.
- ☐ 12. Request for classification on Form P.9.
- ☒ 13. Form P.2 in duplicate.
- ☐ 14. Other.

74 ADDRESS FOR SERVICE: SPOOR &amp; FISHER, SANDTON

Dated: 20 February 2002


SPOOR & FISHER  
PATENT ATTORNEYS FOR THE APPLICANT(S)

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REPUBLIC OF SOUTH AFRICA  
PATENTS ACT, 1978

## PROVISIONAL SPECIFICATION

(Section 30(1) – Regulation 27)

OFFICIAL APPLICATION NO.

LODGING DATE

21	01	2002/1428
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22	19 FEBRUARY 2002
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FULL NAMES OF APPLICANTS

71	DE BEERS INDUSTRIAL DIAMONDS (PROPRIETARY) LIMITED ELEMENTSIX (PTY) LTD 27.01.03 NAAM VERANDER NAME CHANGED
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FULL NAMES OF INVENTORS

72	PIPKIN, NOEL JOHN
----	-------------------

TITLE OF INVENTION

54	METAL COATED ABRASIVE PARTICLES
----	---------------------------------

## **BACKGROUND OF THE INVENTION**

This invention relates to metal coated abrasive particles, or grit, and more particularly to coated grit with improved retention and resistance to oxidation in saw blade segments.

Abrasive grits such as diamond and cubic boron nitride particles, are widely used in sawing, drilling, grinding, polishing and other abrasive and cutting applications. In such applications the grits are generally surrounded by a matrix consisting of metals such as Fe, Co, Ni, Cu and alloys thereof (metal bonds). Alternatively, resin (resin bond) or vitreous (vitreous bond) matrices can be used, the choice of matrix being a function of the particular application in which the abrasive is to be used.

Coating diamond with metals consisting of the Group IVa, Va and VIa transition metals (Ti, Zr, Hf, V, Nb, Ta, Cr, Mo and W) or alloys thereof, and/or their respective carbides, has been shown to improve the performance of abrasive grits. In particular, coated diamonds are used extensively in metal bond applications such as sawing and drilling.

The methods for depositing the metal layers on abrasive grits include PVD methods such as described in "Vacuum Deposition of Thin Films" by L. Holland, Chapman and Hall, 1<sup>st</sup> Edition 1956. Vapour phase CVD methods such as described by M J Hampden-Smith and T T Kodas in "Chemical Vapour Deposition", Vol. 1, No. 1, 1995 can also be used. Alternative CVD methods involve the mixing of the abrasive grits with oxidised metal powders and heating under inert atmosphere (usually vacuum) such as described by

V G Chuprina (Soviet Powder Metallurgy and Metal Ceramics 1992, Vol. 31, No. 7, pp 578-83 and ibid 1992, Vol. 31, No. 8, pp 687-92) or the mixing of abrasive grits and metal powders and heating in a halide (fluorine, chlorine, iodine and bromine or hydrogen compounds thereof) containing inert gas such as described in ASTM B874-96 Standard Specification for Chromium Diffusion Coatings and ASTM B875-96 Standard Specification for Aluminium Diffusion Coatings. These latter processes are often referred to by the term "Pack Cementation". The use of molten alkaline metal halides such as described by T T Oki and Tanikawa in Proceedings of 1<sup>st</sup> International Conference on Molten Salt Chemistry and Technology, p 265, 1983 also offers a means of coating diamonds with the Group IVa, Va and VIa transition metals. This latter method uses a similar chemistry to that of the CVD methods cited and all of these chemical methods are considered in the following description under the heading CVD.

#### **SUMMARY OF THE INVENTION**

According to a first aspect of the invention, a method of producing a metal coated abrasive particle includes the steps of activating a surface of the particle by creating on the particle a suitable number of nucleation growth sites for a transition metal selected from zirconium, hafnium, niobium, tantalum, tungsten and molybdenum and depositing a layer of such transition metal on the activated surface.

The creation of the nucleation growth sites is preferably achieved by use of a carbide-forming metal selected from titanium, vanadium and chromium. Chromium is the preferred activation metal.

The invention has particular application to the coating of diamond particles.

In one preferred form of the invention, a sufficient quantity of the activation metal is deposited on the surface of the diamond to create nucleation growth sites, each nucleation growth site being a particle or region of the carbide of the activation metal. Such sites or regions preferably do not cover the entire surface of the diamond.

The transition metal which is deposited on the activated surface will cover the surface completely and will, in the case of diamond, form a carbide with the diamond surface. The outer surface of the transition metal layer may be metallic in nature.

In another form of the invention, an abrasive particle is placed in a mass of the transition metal, in particulate form, such mass containing a small quantity of activation metal, and the mass and particle subjected to heat treatment under conditions suitable to allow creation of nucleation sites on the diamond surface and coating of the activated surface by the transition metal. The heat treatment should take place under conditions at which degradation of the abrasive particle, e.g. graphitisation in the case of diamond, is minimised.

The heat treatment, in the case of diamond can and preferably takes place in the form of a gaseous halide, particularly gaseous chloride. The gaseous halide can be produced *in situ* by providing the mass and particle with a halide which volatilises under the conditions of heat treatment.

The diamond particles are preferably those suitable for saw applications and may be blocky and strong in nature. Such particles will generally have both cube {100} facets, and/or octahedra {111} facets. Such particles will generally have a particle size of at least 170 microns.

## DESCRIPTION OF EMBODIMENTS

The invention will now be illustrated by the following examples.

### **Example 1**

5gm of 595-420 micron De Beers SDB1100 diamond grit was mixed with 20 gm of tantalum powder and 0.024 gm of ammonium chloride ( $\text{NH}_4\text{Cl}$ ). The mixture was encapsulated in nickel and heated to  $900^\circ\text{C}$  in an argon atmosphere and held at this temperature for four hours before cooling to ambient temperature.

The grit was recovered from the mixture by sieving and it was found that the diamond was totally uncoated.

### **Example 2**

The same procedure as described in Example 1 was followed but with 0,01 wt% chromium powder mixed with the tantalum powder.

On recovery of the diamond grit after, the heating cycle, it was noted that all of the diamond particles were partially coated with metal which was shown by X-ray diffraction methods to be tantalum carbide.

It was noted that preferential growth of the tantalum carbide occurred on the {100} or cube crystal facets.



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### Example 3

The same procedure as described in Example 2 was followed but a suite of samples were prepared containing 0,05 wt% Cr, 0,10wt%, 0,20 wt%, 0,50 wt%, 1,0 wt% and 2,0 wt% chromium powder mixed with the tantalum powder.

On recovery of the diamond grit, after the heating cycle, it was noted that all of the diamond particles were coated with a coherent dense layer of metal which was shown by X-ray diffraction methods to be tantalum carbide.

It was a further feature of these coatings that the thickness of the chromium carbide layers increased as the chromium concentration in the starting mixtures increased to the highest level of chromium (2 wt%). At these higher chromium levels there is a tendency for the coating to crack and spall off.

It is thus preferred that as little chromium as is necessary to induce the deposition of a coherent tantalum carbide layer is used.

### Example 4

The effect of temperature and time on the coating rate was examined using tantalum powder containing 0,1 wt% Cr chromium powder.

The following table shows the temperatures and heating times used and the mass of coating measured. The coating mass is the average mass of the coating expressed as a percentage of the mass of the coated particles.

Temperature (°C)	Time (hrs)	Coating Mass (wt%)
900	4	3,12
900	1	2,34
850	4	2,33
800	4	1,99

On recovery of the diamond grit, after the heating cycles, it was noted that in all cases the diamond particles were coated with a coherent dense layer which was shown by X-ray diffraction methods to be tantalum carbide.

#### Example 5

6 gm of 595-420 micron De Beers SDB1100 diamond grit was mixed with 8 gm of niobium powder and 0,024 gm of ammonium chloride ( $\text{NH}_4\text{Cl}$ ). The mixture was encapsulated in nickel and heated to 850°C in an argon atmosphere and held at this temperature for four hours before cooling to ambient temperature.

The grit was recovered from the mixture by sieving and it was found that the diamond was only sparsely coated. It was noted that the {100} cube facets coated more readily than the {111} octahedral facets.

#### Example 6

The same procedure as described in Example 5 was followed but a suite of samples was prepared containing 0,01 wt% Cr, 0,05 wt%, 0,01 wt% and 0,20 wt% chromium powder mixed with the niobium powder.

On recovery of the diamond grits those coated using the 0,01 and 0,05 wt% showed incoherent coating whereas the two high chromium additions resulted in grits which had totally coherent coatings. The following table lists the coating data for these grits.

Chromium addition (wt%)	Coating mass (wt%)
0,01	0,68
0,05	0,72
0,1	3,66
0,2	2,87

**Example 7**

6 gm of 595-420 micron De Beers SDB1100 diamond grit was mixed with 10 gm of tungsten containing 0,08 wt% chromium and 0,024 gm of ammonium chloride ( $\text{NH}_4\text{Cl}$ ). The mixture encapsulated in tantalum and heated to  $900^\circ\text{C}$  in an argon atmosphere and held at this temperature for four hours before cooling to ambient temperature.

On recovery of the grit it was found that the diamond cube facets were coated and the octahedral facets partially coated.

This result can be compared with Example 1 and shows that the chromium present in the tungsten powder has enhanced the nucleation of the tungsten carbide on the diamond grit.

Dated this 20<sup>th</sup> day of FEBRUARY 2002

  
.....  
Spoor & Fisher  
Applicant's Patent Attorneys

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